# INTERFACE CONFIGURATION EXPERIMENT PRELIMINARY RESULTS

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# **ABSTRACT**

The Interface Configuration Experiment (ICE) was carried out on USML-1 to investigate liquid-gas interfaces in certain rotationally-symmetric containers having prescribed, mathematically derived shapes. These containers have the property that they admit an entire continuum of distinct equilibrium rotationally-symmetric interfaces for a given liquid volume and contact angle. Furthermore, it can be shown that none of these interfaces can be stable. It was found, after the containers were filled in orbit, that an initial equilibrium interface from the symmetric continuum reoriented, when perturbed, to a stable interface that was not rotationally symmetric, in accordance with the mathematical theory.

#### INTRODUCTION

It is essential, when planning space-based operations, to be able to predict the configurations that fluids will assume in their containers under low-gravity conditions. For example, one would be in serious difficulty if one did not know in advance in what part of a fuel tank the liquid contents were to be found. Currently available mathematical theory can determine possible free-surface configurations for only a few simple containers, such as the right circular cylinder and the sphere. Even for these geometries, the present theory is incomplete for dealing with unaccustomed liquid configurations, such as a column or bridge extending across the interior of the container, that can be expected to occur under reduced gravity.

The classical theory, according to the Young-Laplace-Gauss formulation, characterizes stable fluid configurations as local minima of the surface-plus-gravitational mechanical energy. Based on this point of view we have been carrying out mathematical studies directed toward characterizing possible configurations of liquids under reduced gravity. In this connection we have calculated the shapes of rotationally symmetric "exotic" containers with the remarkable property that for given contact angle and

liquid volume, an infinity (in fact, an entire continuum) of distinct rotationally symmetric equilibrium configurations can appear, all of which have the same energy (Figure 1).<sup>5,10</sup> These symmetric equilibrium configurations turn out to be unstable, and it can be shown that particular deformations that are not rotationally symmetric yield configurations with lower energy.<sup>4,10</sup> By a careful construct, using a result of Ref. 11, it was possible to demonstrate a symmetric container that admits infinitely many symmetric equilibrium interfaces, but for which no interface that minimizes energy can be symmetric. This is in notable contrast with what happens in the familiar case of the right circular cylinder, for which the symmetric interface is stable, and no asymmetric ones can appear.<sup>13</sup>

There is presently no known way to determine mathematically the surfaces that minimize energy in the exotic containers. Numerical computations have suggested a number of particular non-rotationally-symmetric surfaces as local minima. As it is not clear to what extent the idealizations of the formal theory will be reflected in reality, it is of interest to determine experimentally what physically observable energy-minimizing configurations there might be. Our USML-1 experiment has been carried out for this purpose, both as an indication of the limitations of classical theory and as a guide to future theoretical study. A preliminary form of this paper is given in Ref. 8.

# I. COMPUTED CONFIGURATIONS

According to the classical Young-Laplace-Gauss formulation, the mechanical energy  $\epsilon$  of a partly filled container, arising from the associated surface and hydrostatic forces, is (Ref. 9, Chap. 1)

$$\varepsilon = \sigma(S - S^* \cos \gamma) + \text{gravitational energy}$$
 (1)

Here  $\sigma$  is the liquid-gas interfacial tension and  $\cos\gamma$  is the liquid-solid relative adhesion coefficient, where  $\gamma$  is the contact angle between the liquid and the container; S denotes the area of the liquid-gas free surface and S\* the area of the portion of the container in contact with the liquid. The quantities  $\sigma$  and  $\gamma$  depend only on the materials and are independent of the container geometry and gravitational field. The classical formulation, upon which our mathematical study is based, does not encompass possible surface friction effects, such as hysteresis and contact line resistance, or thermodynamic Kelvin energy associated with evaporation and condensation. Configurations of liquid that yield a stationary value for  $\varepsilon$  subject to the constraint of fixed liquid volume are, according to the classical theory, equilibrium configurations. They will be stable, as well, if  $\varepsilon$  is locally minimized. In Ref. 5 exotic rotationally symmetric container shapes are calculated from (1) and the requirement that there be a continuum of rotationally symmetric liquid-gas free surfaces all having the same contact angle and enclosing the same volume of liquid with the container. Such container shapes, which were studied first in Ref. 12 for the

special case of zero gravity and contact angle  $\pi/2$ , can be obtained for any contact angle or gravity level.<sup>5,10</sup>

The container shown in Figure 1 is obtained by placing circular cylindrical extensions with disk ends on a calculated exotic container shape, in this case for zero gravity and contact angle 80°. The dashed curves depict members of the continuum of rotationally symmetric equilibrium interfaces. Although such containers can be constructed for any gravity and contact angle, only under microgravity conditions are they of sufficiently large scale to permit accurate physical experiment and observation. Of principal interest in this study is the case for which gravity is zero, or sufficiently small that the gravitational energy term can be neglected, so that (1) becomes

$$\varepsilon = \sigma(S - S^* \cos \gamma).$$

In order to obtain an indication of what stable interfaces there might be in an exotic container, such as the one in Figure 1, numerical calculations were carried out using a modified version of the Surface Evolver program<sup>1</sup> and the driving software reported in Ref. 2. The Surface Evolver seeks local minima of a discretized energy functional subject to prescribed constraints using a gradient descent method. Surfaces are approximated by a piecewise-linear triangulation, the form of which can be controlled to various degrees with commands available to the user. Under control of the user, the program adjusts the triangulated surface, step-by-step, in an attempt to decrease the energy. From the numerical and graphical output provided by the program, a user interprets whether a local minimum has been found.

Numerical results obtained with the Surface Evolver for the configuration depicted in Figure 1, but with container shape and liquid for contact angle 55°, are shown in Figure 2. (The value of 55° corresponds to the materials for which the USML-1 video images are shown below in the Video Images section). The computed surface depicted in Figure 2 is the lowest-energy one that was found using the Surface Evolver, starting from the initial horizontal planar member of the symmetric equilibrium interface continuum, to which a small volume-preserving perturbation had been added. Other local minima could be found by varying the form of the initial perturbation, but these had greater energy, although still less than that for the rotationally symmetric surface continuum.<sup>2</sup> The minimizing free surface is shown as a lighter surface against the darker, checked background of the container; both have been shaded by the (color) graphics display software. Triangulation mesh lines are in white. To expose the free surface to view, only half the container, lying to one side of a vertical plane through its axis of symmetry, is shown. This vertical plane is one about which the free surface possesses reflective symmetry. The exotic, bulge portion of the container and a small length of the circular cylindrical extensions are depicted. The view

for the upper figure is from a point on a horizontal plane through the maximal width of the bulge; the vertical symmetry plane of the free surface is in the plane of the paper. The liquid would lie below and to the left of the free surface. For the lower figure the viewpoint is slightly above the maximal-bulge plane and to the right of the one for the upper figure, to show the excursion of the surface across the bulge portion of the container.

# II. EXPERIMENTAL APPARATUS

The vessels for the Interface Configuration Experiment, one of which is depicted in Figure 3, were fabricated at the NASA Lewis Research Center. Each weighs about 1.5 Kg, including the liquid contents, and measures approximately  $9 \times 9 \times 18$  cm. The interior was bored out of a solid rectangular block of acrylic plastic, to limit optical distortion. The coordinates for the bulged portion of the container were fed into a numerically controlled air bearing lathe, which performed the final machining operations. The surfaces were then lightly finished with cloth and a polishing compound. After polishing and annealing, the interior surface deviated less than 50  $\mu$ m from the specified, calculated one.

The vessels were designed for use in the USML-1 Glovebox Experiment Module, which was provided by the European Space Agency/ESTEC. The Glovebox is a multi-user facility developed for experiments to be conducted on Spacelab missions beginning with USML-1. The purpose of the Glovebox is to provide a work area that is ergonomically sound and will allow a payload specialist to carry out operations using small quantities of toxic, irritant, or potentially infective materials, which must be prevented from contaminating the spacecraft atmosphere. Previously designed to handle biological experiments, the Glovebox has been adapted to handle fluids, combustion, and materials science experiments, to permit effective use of hands-on interaction by the payload specialists. ICE utilized the Glovebox primarily as a staging area and a level of containment in the event of fluid leaks. ICE was one of sixteen Glovebox experiments aboard the NASA Space Shuttle Columbia on USML-1 (STS-50).

# III. EXPERIMENT DESCRIPTION

The general experimental procedure for ICE during the USML-1 flight was to partly fill the selected vessels with prescribed volumes of fluid and to record with two video cameras the fluid interface configurations that resulted.

Four vessels were fabricated for the tests. They are similar in construction to the vessel shown in Figure 3. The primary vessel components are the single-piece acrylic-plastic (transparent) body, an aluminum piston and control dial, stainless steel drive screw and two-port valve, and magnetized feet for securing the vessel to the Glovebox labjack. As indicated in Figure 3, the vessel is placed in the Glovebox in a horizontal position. One of the test fluids is an "immersion" fluid that has a refractive

index matched with that of the acrylic container, for reduction of optical distortions; it is a blend of hydrogenated terphenyl and an aliphatic hydrocarbon. Two of the vessels contained this fluid. The interiors of these vessels were coated with surface modifier FC-723 to produce a desired contact angle. Distilled water was used in the other two containers, which were not coated.

The fluids were lightly dyed in order to enhance visibility of the free surface. For the immersion fluid a ceres red dye was used, and for the water a blue food coloring was used, which was an aqueous solution of propylene glycol with propylparaben and sodium metabisulfite as preservatives. Both concentrations were so small that no measurable effects were detected on the values of surface tension or contact angle after the dye was introduced. Surface tension was measured by the Dunuoy ring method as 72.4 dyne/cm for the water and 32.4 dyne/cm for the immersion fluid, with a repeatable accuracy of  $\pm$  0.2 dyne/cm. The wedge method (Ref. 3, pp. 220 - 221), (Ref. 6, pp. 191 - 192), was used for measuring the contact angle of water with the acrylic plastic as 80° with a repeatable accuracy of  $\pm$ 2°. For the immersion fluid, a modified sessile drop method yielded a contact angle with the coated surface of approximately 55°. This method is more sensitive to hysteresis effects; the accuracy is estimated at  $\pm$ 4°.

The interior surfaces of each vessel were cleaned with sequential rinses of a strong ethanol/distilled water solution and distilled water. The vessel was then allowed to dry in a pure nitrogen clean-room environment. Before being injected into the vessel reservoir, the fluid was filtered with P4 filter paper. The injection was carried out in the nitrogen environment. All seals on a vessel were lubricated with the particular fluid used for it, to eliminate possible contamination from other lubricants.

The two immersion-fluid-filled vessels were the same, the exotic container portion being constructed for the 55° contact angle measured for the materials; the second provided a repeat run for experiment control purposes. For the third vessel, which contained distilled water, the exotic container portion was constructed for the contact angle of 80°. A similar vessel was tested previously in the NASA Lewis Research Center Zero Gravity Facility five-second drop tower. Although insufficient low-gravity time was available for a stable interface to be achieved, observations showed reorientation toward a particular asymmetric configuration. The 80° vessel tested on USML-1 permits comparison with these ground-based test results, and, more importantly, for obtaining information on the behavior resulting from disturbing the surfaces that form in space.

The fourth vessel also contained distilled water as test fluid, yet it differs from the others in that the container is not exotic; the bulge is a portion of a sphere, for which complete mathematical results are available. The infinite family of distinct rotationally symmetric equilibrium interfaces that are possible for the exotic containers cannot form. However, by rigid-body rotation of the liquid, an equilibrium surface in zero gravity can be tilted as desired with respect to the symmetry axis, without any change in

shape or energy. The results for this vessel can act as a control to compare with the behavior in the exotic containers.

The diagnostics for the experiment included two full-color 1:1 video cameras to record the fluid interface configurations, a Glovebox video camera with audio, and devices for the measurement of ambient Glovebox temperature and local acceleration levels.

# IV. EXPERIMENT PROCEDURE

The crew procedures for carrying out ICE consisted of six steps: (1) unstow equipment, (2) set up Glovebox and install vessel, (3) fill vessel test volume with fluid, (4) observe equilibrium interfaces, (5) disturb interface configuration(s) to determine stability, (6) reverse fill procedure and stow. Up to 60 minutes was required for each vessel when it was tested independently. Less time was necessary when the vessels were tested sequentially.

To begin the experiment, a crew member unstowed the labjack, video cameras, multi-use arm and clamp, and the ICE vessel to be tested. The Glovebox power was switched on and the ICE vessel was placed (horizontally) on the labjack. One video camera was mounted to the front door of the Glovebox, and the other was directed down from the top, secured by the multi-use arm and clamp. The field of view for the cameras was centered on the bulge portion of the exotic container. Iterative adjustments of the cameras and vessel were necessary to center the field properly for both cameras. The Glovebox doors were then replaced with cuff attachments, and the cameras were turned on to record the interface configuration.

To carry out the fill procedure, the crew member opened the quarter-turn valve and turned the control dial on the vessel, displacing the entire fluid contents of the reservoir slowly into the exotic container. The volume of fluid corresponded to that for the symmetric family of equilibrium interfaces, as calculated for the planar member of the family (Figure 1). Time was then allowed for the fluid configuration to stabilize fully while being recorded by the cameras. Five minutes were allowed for the filling procedure. The crew member then disturbed the surface by tapping the container with a finger, lightly at first and then subsequently with moderately increasing force. New surfaces that formed in the container during the tapping procedure were given time to stabilize and to be captured on video. The tapping continued until the surface either broke up or consistently returned to a particular configuration, at which point the fill procedure was to be reversed to empty the exotic container test volume. The vessel and support equipment were then stowed.

#### V. VIDEO IMAGES

Because the Glovebox video recordings of ICE taken on board USML-1 started to be released only recently, there has not yet been opportunity for a complete analysis. However, frames from the videotapes shown in Figures 4 and 5 indicate qualitative behavior of the liquid for one of the 55° exotic containers. For the other exotic containers, the behavior was found to be similar.

Figure 4 shows views taken with the video camera that was directed downward from the top of the glovebox. The views correspond to looking down on the module and schematic of Figure 3 with the viewer behind the module, so that the container portion is on the left and piston and dial on the right. The first frame shows the equilibrium configuration after filling has been completed. This would correspond to the member of the continuum depicted in Figure 1 that is uppermost at the container axis. The subsequent sequence of four frames shows the reorientation following a perturbation resulting from payload specialist Lawrence DeLucas tapping the dial end of the module. The last frame depicts the configuration after the fluid again reached equilibrium. This last configuration was observed to be very stable to subsequent perturbations that were applied - even to those that were relatively large.

Figure 5 depicts the initial and final equilibrium shapes as viewed from the video camera mounted to the front door of the glovebox. Here the (dyed) liquid is better illuminated than in Figure 4, because the glovebox back-lighting is directly behind the liquid relative to the camera. Although the shadows and reflections make it difficult to discern the detailed shape of the final equilibrium interface in the black-and-white Figures 4 and 5, it is evident from the figures that the fluid reorients to a non-rotationally-symmetric shape, not unlike the computed lowest energy configuration shown in Figure 2.

## VI. POSTFLIGHT DATA ANALYSIS

Taking into account video recording excerpts downlinked to Earth during the flight and comments made by the payload specialists, some qualitative remarks can be made. In all cases, the displacement of liquid from the reservoir to the container was reported as being accomplished successfully, and at the end of the filling procedure the observed free surface was generally in accordance with one that was predicted: a spherical cap corresponding to a particular member of the continuum of rotationally symmetric (unstable) equilibrium interfaces. The liquid then moved to an asymmetric configuration in the each of the three exotic containers after it was tapped by the payload specialist, in a manner similar to that shown in Figure 4. This is in accordance with mathematical predictions, and the shapes appeared to be in correspondence with the computed lowest energy configurations. The asymmetric configuration behaved very stably in response to subsequent induced disturbances. No configurations resembling those from other computed local-minimum energies at higher

energies were observed. The interface in the control vessel with spherical bulge did not exhibit the type of reorientation described above; this, too, is in accordance with theory.

Corroboration and quantitative evaluation of the results, as well as further assessment of effects of hysteresis and other factors, await complete analysis of the data.

#### VII. CONCLUDING REMARKS

The Interface Configuration Experiment (ICE) on the first NASA United States Microgravity Laboratory (USML-1), launched in June 1992, explored a striking behavior of liquid-vapor interfaces that has been predicted mathematically for certain "exotic" containers in a low-gravity environment. Results from video images show that an initial equilibrium interface from the rotationally symmetric continuum reoriented, when perturbed, to a stable interface that was not rotationally symmetric, as predicted by the idealized mathematical theory. The results indicate also the role of contact-line resistance forces (contact-angle hysteresis)in creating a barrier that must be overcome, in moving from the symmetric equilibrium configuration into asymmetric configurations of lower potential energy. More detailed and extensive experiments can be expected to yield further information on the significance of the findings and on sensitivity to effects not included in the present theory, such as those associated with contact-line resistance and with inaccuracies in measuring contact angles and in fabricating the vessels. The present and future experiments can contribute to determining the applicability of the existing theory and the ability of the theory to predict fluid interfacial configurations for arbitrary container geometries.

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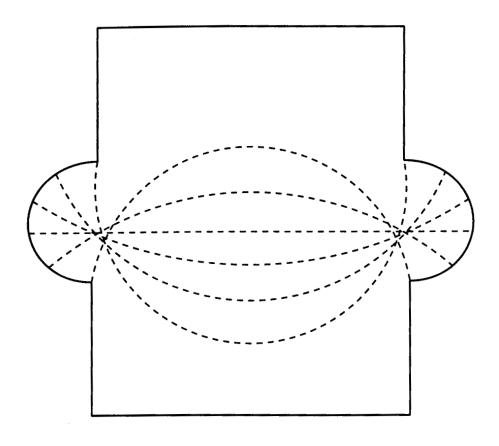
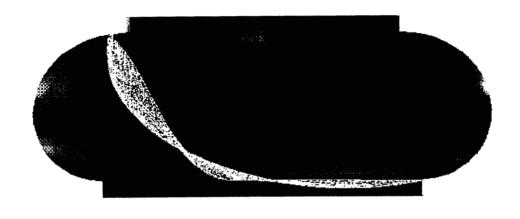


Figure 1 Axial section of an exotic container for contact angle 80° and zero gravity, depicting meridians (dashed curves) of members of the rotationally-symmetric equilibrium free surface continuum. All surfaces have the same contact angle and energy and enclose the same volume of liquid with the bottom of the container.



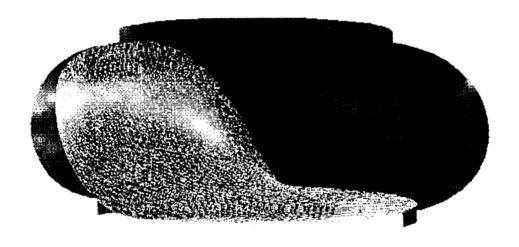
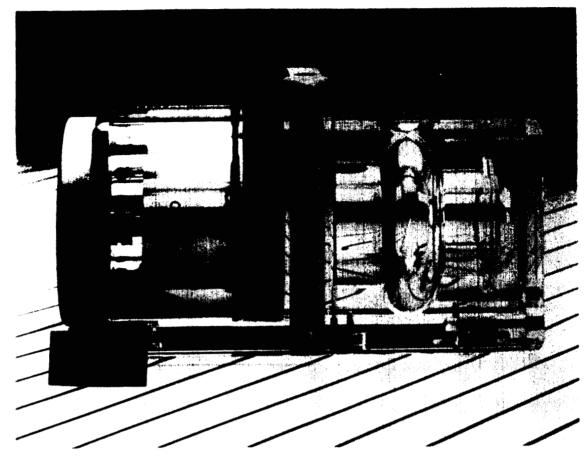


Figure 2 Two views of calculated energy-minimizing equilibrium interface in an exotic container. Zero gravity,  $\gamma = 55^{\circ}$ .



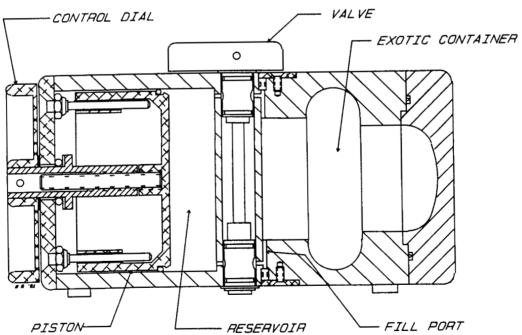


Figure 3 Flight module and schematic drawing of an interface configuration experiment vessel. Exotic container is for zero gravity and zero gravity,  $\gamma = 55^{\circ}$ .

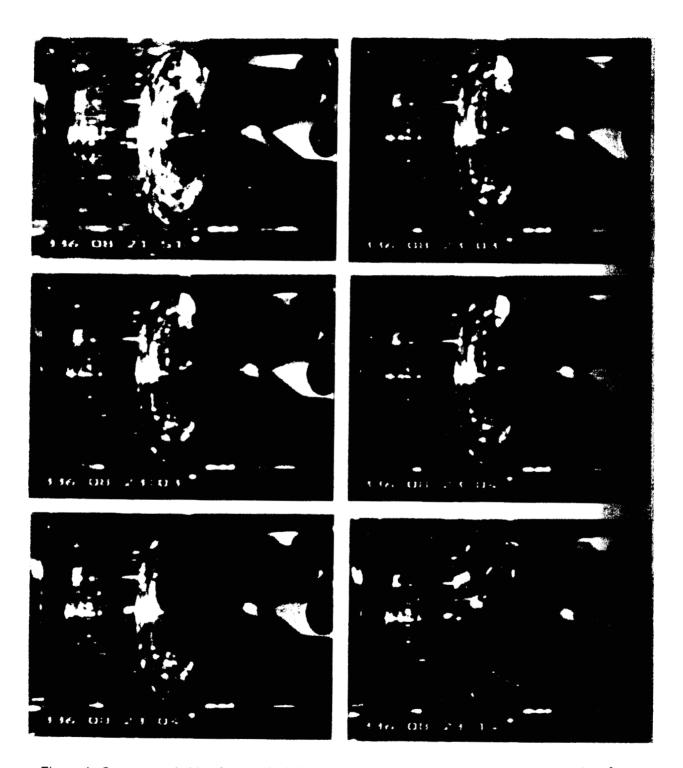


Figure 4 Sequence of video frames depicting transition from initial symmetric equilibrium interface (upper left) to stable asymmetric one (lower right). Top view.

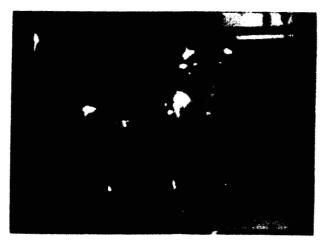




Figure 5 Initial symmetric equilibrium interface (left) and stable asymmetric one (right). Front view.

# Discussion

Question: Were any other orientations tried in the experiment?

Answer: No. Everything was done in the Spacelab in the Glovebox with the vessel lying horizontally. That is the way it fit into the Glovebox and fit onto the lab jack. It had magnetic holes in the bottom, so there was no attempt to orient the container in any other way. Of course if there is no gravity at all, it wouldn't matter. There is some.

Question: Were there any noticeable differences between the two runs you showed?

Answer: This is the second run. The first run, I believe, Larry tapped the vessel from the side, so it was an asymmetric perturbation which would encourage this configuration you saw on the slides here. The second one you saw was tapped on the end in an attempt to get a symmetric perturbation which would be less biased towards having the fluid move that way (as in the first run). But it always went to something like that.

Question: Have you tried to correlate your results to SAMS data?

Answer: No. We would like to get the SAMS data and put it up along side to see if we could observe something. That is certainly on the list once we get all the tapes.

Question: What is your next experiment on the lines shown here?

Answer: We have something that has been proposed and accepted for USML-2. But whether it is still on it we do not know, since the Glovebox is over-subscribed. It is actually a different sort of phenomenon. It is one of discontinuance dependence on configuration and contact angle whereby a small change in the container geometry or a small change in contact angle could cause large bulk motions of the fluid. We want to study that and possible applications; by which I mean determining contact angles in zero gravity in an accurate way.